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ALL-UNION INTER-VUZ CONFERENCE ON THE THEORY  
AND PRACTICE OF RECTIFICATION IN THE CHEMICAL  
AND THE FOOD INDUSTRIES

by P. Tsygankov

- USSR -

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ALL-UNION INTER-VUZ CONFERENCE ON THE THEORY  
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- USSR -

Following is the translation of an article by P. Tsygankov in Izvestiya Vysshikh Uchebnykh Zavedeniy SSSR, Khimika i Khimicheskaya Tekhnologiya (Bulletin of Higher Educational Institutions USSR, Chemistry and Chemical Technology), Vol IV, No 2, Ivanovo, 1961, pages 328-332.

The All-Union Inter-Vuz Vysshiye Uchebniye Zavedeniya Conference was held 24-29 October 1960 in the Kiev Technological Institute of the Food Industry, examining basic problems of the theory and practice of the process of rectification in the chemical and the food industries. The conference carried out the coordination of studies of numerous establishments working in the field of rectification and outlined the future approaches to the organization of scientific research studies into rectification.

Participating in the work of the conference were 230 delegates, representing 27 higher and secondary specialist educational establishments, 41 scientific research organizations, and 25 industrial enterprises. At the conference 64 papers were read, which gave a full accounting of the directions of studies in various establishments participating in the work of the conference.

In the first report Prof V. V. Kafarov, of the Moscow Chemico-Technological Institute imeni D. I. Mendeleyev (MKhTI) Moskovskiy khimiko-tehnologicheskiy institut imeni D. I. Mendeleyeva, outlined the most important problems in the modern status of the theory of rectification processes: equilibrium of phases in the rectification process; determination of static parameters in the rectification process; and kinetic parameters of the rectification process.

Prof V. N. Stabnikov, of the Kiev Technological Institute of the Food Industry (KTIPP) /Kievskiy Tekhnologicheskiy Institut Pishchevoy Promyshlennosti/, in his paper stated the new directions in the designing of rectification apparatus. The creation of high-productivity small-scale rectification apparatus -- possessing high separating power -- is the basic task confronting designers. The development of rectification apparatus is proceeding in the following directions: improvement of contact units of plate columns; the improvement of packed towers; the development of film apparatus; and the design of high-efficiency rotation apparatus.

In conclusion Prof V. N. Stabnikov dwelt on schemes making possible the realization of a high economy in energy consumption in rectification assemblies.

Prof P. A. Semenov, of the Moscow Institute of Chemical Machine Building (MIKhM) /Moskovskiy Institut Khimicheskogo Mashinostroyeniya/ outlined the problems of rapid /skorostniy/ mass exchange, which is basically grounded on the increase in velocity in the vapor phase, the maximum increase in the frictional portion of the hydraulic mass exchange, and the replacement of counter-current motion of the phases by direct-stream. In rapid apparatus the local velocity of the vapor stream can attain values up to 50-80 m/sec; in relation to the complete cross-section of the apparatus, up to 8-10 m/sec.

Docent Yu. V. Tumanov, of the MIKhM, reported on results of a study of one of the apparatus containing rapid mass exchange -- a non-sprayer apparatus with irrigated /oroshayemyy/ Venturi tube. The use of this apparatus for the rectification of liquid air has made possible the attainment, relative to the apparatus cross-section, of the order of 5 m/sec. Docent Yu. V. Poplavskiy, of the MIKhM, reported on the designing and calculations for plate rectification columns with straight current contact assemblies, in which rapid mass exchange has also been realized. The introduction of columns with straight flow contact assemblies has made possible an increase by 10 to 15 times of the volume product per unit volume of the apparatus. Graduate student A. P. Sinkha, of the MIKhM, reported on the effect of surface-active substances on rapid mass exchange.

A large number of reports were devoted to study of grid /reshetchatyy/ plates of the saddle /probal'nyy/ type.

Scientific worker N. A. Kochergin (V. M. Olevskiy and V. V. Dil'man) of the State Institute of the Nitrogen Industry (GIAP) /Gosudarstvennyy Institut Azotnoy Promyshlennosti/ made a report about a study of mass transfer in perforated saddle /dyrchatyye proval'nyye/ plates with an

active Zhiviy cross-section of 15-25 % and interstices 2-4 mm, under the conditions of the rectification of 28 organic mixtures.

A paper by scientific worker N. I. Zelentsovaya (M. E. Aerov, T. A. Bystrov, Ye. P. Darovskiy, and A. A. Kovalev), of the Scientific Research Institute for Synthetic Alcohol (NIISS) Nauchno-Issledovatel'skiy Institut Sinteticheskogo Spirta, was devoted to a study of the hydraulics, mass and heat exchange on grid plates of the saddle type in the presence of two liquid phases. It was established that the stratified liquids on grid plates intermingle well, not forming stable emulsions. The efficiency of the plates was obtained as a function of the load.

Scientific worker K. A. Kalunyants (G. I. Fertman), of the Central Scientific Research Institute of the Alcohol Industry (TsNIISP) Tsentral'niy Nauchno-Issledovatel'skiy Institut Spirtovoy Promyshlennosti, made a report on the extended action of grid plates of the saddle type in the fractional distillation of raw alcohol. The experiments indicated that fractional distillation proceeds with the same measure of success also on cap plates, but however, the alcohol volume is increased by two to three times.

Scientific worker L. Ye. Sum-Shik (M. E. Aerov and T. A. Bystrov), of the NIISS, reported on results of a study on the take-up Unos of liquid on grid plates of the saddle type. It was indicated that take-up on the grid plates is 1.5-2 times less than on cap plates.

Researcher Engineer D. N. Popov (A. G. Kasatkin and Yu. I. Dytcherskiy), of the MKhTI, reported on the hydraulic testing of plates and the derivation of a formula for the permissible gas velocity in using grid plates of the saddle type. He showed that the height of the foam is a basic parameter determining the hydrodynamics and efficiency of mass transfer.

The contribution of researcher Engineer I. F. Malezhik (V. N. Stabnikov), of the KTIPI, was concerned with the study of the performance of a new type of contact assembly -- valve plates klapannyye tarelki. In studying the hydrodynamics and efficiency of the valve plates with alcohol-water mixtures the speaker drew a conclusion on the expediency of the application of this apparatus for rectification purposes in the alcohol industry. Valve plates have high significance in terms of efficiency and permit greater vapor velocity.

Worker A. S. Vigdorov (V. V. Kafarov and L. I. Blyakhman), of the State Scientific Research Institute of Organic By-Products and Resins (NIOPiK) Gosudarstvennyi Nauchno-Issledovatel'skiy Institut Organicheskikh Poluproduktov

i Krasiteley<sup>7</sup> made a report on a new design on an overflow assembly of plates, securing the maintenance of a high foam level on the plates and also favorable conditions of overflow.

Chief Engineer of the project "Giproneftemash" /Gosudarstvennyy Nauchno-Issledovatel'skiy i Proyektnyy Institut Neftyanogo Mashinostroyeniye -- State Design and Scientific Research Institute for Petroleum Machinery/, V. A. Sheynman (Ya. I. Frenkel', Yu. S. Kogan, A. S. Fandeyev, I. F. Babitskiy, M. A. Berkovskiy, Yu. I. Vol'shonok, and V. A. Lizunkov) reported on a study dealing with the creation of high-productivity rectification columns. On a cold stand in a system of air-water it was established that fluted plates perform with a productivity 20-30 % lower than for cap plates; the plates "Uniflyuks" correspond in productivity to cap plates, but they are simpler in design and are lighter and easier to assemble. Good results have been obtained by plates filled with gapped exhaust sheet /prosechniy vytyazhniy list<sup>7</sup>/ with an active cross-section of 22%, and with recoil elements from the same kind of sheet, positioned obliquely at a spacing of 200 mm one from the other. The plate has a productivity of 2.5-3 times higher than that for cap plates.

Prof L. S. Aksel'rod, of the MIKhM, reported in his paper that in rectification columns containing screen plates /sitchatyye tarelki/ in assemblies strongly chilled /ustanovok glubokogo kholoda/ high velocities of 0.7-0.65 m/sec, with an inter-plate distance not less than 120 mm and a vapor density 5-30 kg/m<sup>3</sup>, are attainable, which are the maximum velocities attainable. Then the speaker indicated the need for using experimental data in calculations of the value of plate efficiency and also directed attention to the economy of power consumption, which guarantees a column performance, having a surplus flux of 10-15 %.

Graduate student O. S. Chekhov, of the MIKhM, reported on the intensity of mass exchange in bubble plates and on their hydraulic resistance. Worker I. A. Aleksandrov (A. I. Skoblo and Yu. K. Molokanov), of the "Giproneftemash", contributed a paper dealing with the selection of optimum dimensions of plate columns, taking into account the entrainment of liquid by the gas stream.

The problem of the performance and regulation of packed columns was the subject of a contribution by scientific worker L. I. Blyakhman, of the NIOPiK. Docent I. P. Slobodyanik, of the Krasnodarsk Institute of the Food Industry, presented a method of analyzing the performance of packed rectification columns which permits calculation of the effect of the determining parameters within the wide

limits of their variations on the kinetics of the process. Graduate student A. A. Grinevich (A. G. Bol'shakov), of the Odessa Polytechnic Institute, dwelt on methods and results of a study into the determination of the moistened surface in columns containing ring packing.

Scientific worker V. F. Olevskiy (L. A. Pichugin, and Yu. A. Gromoglasov), of the GIAP, reported on the rectification of thermally unstable substances in film plenochnyye columns of continuous operation. The most advantageous type of column for this purpose is the film column in which the necessary vacuum is easily realized and the liquid holdup is reduced to the minimum.

Prof A. N. Planovskiy, of the MIKhM, contributed a paper entitled "Problems of the Application and Calculation of Plate Rectification Columns." Presenting a critical evaluation of calculations for plate columns, the speaker disclosed that the most physically grounded and objective is through the equation of mass transfer, in which the coefficient of mass transfer is related to the working area of the plate. A. N. Planovskiy dwelt on the effect of entrainment and the importance on the selection of the optimum reflux number for the rectification process. In conclusion the speaker indicated the advisability of using higher pressure in the apparatus. A number of contributions (L. S. Aksel'rod, V. A. Malyusov, A. G. Yevstaf'yev, etc.) emphasized that at present there does not exist the opportunity of using the methods proposed by A. N. Planovskiy for engineering calculations.

The report of Docent A. G. Yevstaf'yev, of the MIKhM, dealt with an evaluation of the efficiency of plate-type column apparatus. In the report methods were given for the calculation of plate efficiency, the relationship between point tochechnyy efficiency, efficiency of plate, and efficiency of column, and also a relationship between the number of plates and the number of unit surfaces.

Docent A. A. Noskov (N. V. Ozerova), of the Leningrad Technological Institute imeni Lensoveta, presented criterion functions that he obtained for the calculation of the coefficient of the enrichment of screen rectification plates, which represents the most convenient value for the calculations of plate columns with crossed streams of vapor and liquid.

Co-worker A. S. Vigdorov (V. V. Kafarov), of the NIOPiK, indicated the applicability of thermodynamics of stationary irreversible processes for the calculation of rectification apparatus.

Co-worker E. G. Bergo (M. E. Aerov and K. P. Berezhnaya), of the NIISS, investigating the process of counter-

current condensation and counter-current vaporization indicated that non-adiabatic mass exchange can substantially decrease the energy expenditure.

A number of reports were devoted to the determination of static parameters of the rectification process. Thus, Docent D. D. Zykov (N. B. Kondukov, V. I. Astakhov, and V. Ts. Khlebnikova), of the MIKhM, in his report illuminated the methods for the calculations of the separation of multi-component systems, the selection of optimum conditions for carrying out the separation in a single column and in an aggregate, and indicated the application of computers for calculations of multi-component mixtures.

Scientific co-worker Ye. Ya. Susanov, of the Krasnodarsk Affiliate of the VNIINeft' /Vsesoyuznyy Nauchno-Issledovatel'skiy Institut Nefti -- All-Union Scientific Research Institute of Petroleum/, presented methods of the grapho-analytic calculation of the separation of multi-component systems. Scientific co-worker A. A. Kondrat'yev, of the Ufa Petroleum Institute, devoted his report to the effect of underheating and overheating of the feed in the calculations of the separation of multi-component systems, and the effect of the underheating temperature on the reflux number.

Scientific co-worker N. Kh. Agliulov (G. G. Devyatkh), of the Scientific Research Institute in the Gor'kiy University imeni A. A. Zhdanov, reported on a formula derived by him which gives the function of the separation rate of the distillate and the separatory capacity of a column. The formula was substantiated by experimental data. Scientific co-worker G. L. Motina (M. E. Aerov), of the NIISS, reported on methods of confirmatory calculations of the rectification of ternary stratified mixtures.

The contribution of scientific co-worker V. S. Timofeyev (S. V. L'vov, Ye. A. Gruzdev, N. M. Klimenko, and L. A. Serafimov), of the Moscow Institute of Fine Chemical Technology, was devoted to an investigation of the rectification process and to methods of calculations of the separation of multi-component non-ideal mixtures by a method of separatory vapors. Scientific co-workers V. M. Platonov and B. G. Bergo, of the NIISS, reported on the calculations for the rectification of mixtures using the analog and digital computers "Ural" and "Strela".

A number of reports were concerned with the problems of azeotropic and extractive distillation. Scientific co-worker V. B. Kogan, of the Leningrad Institute of Applied Chemistry (LIPKh) /Leningradskiy Institute Prikladnoy Khimii/, elucidated on several problems of the theory and practice of azeotropic and extractive rectification. The paper of scientific co-worker S. K. Ogorodnikov (V. B. Kogan and

and M. S. Nemtsev), of the All-Union Scientific Research Institute of Synthetic Caoutchouc (VNIISK) /Vsесоуузныy Nauchno-Issledovatel'skiy Institut Sinteticheskogo Kau-chuka/, was concerned with a study of problems of the separation of mixtures of C<sub>5</sub> hydrocarbons by the method of

extractive and azeotropic rectification. It was shown that the most effective admixture for the rectification of a given mixture is dimethylchloramide. The effect of admixtures on the entire range of isomers and homologs of C<sub>5</sub> hydrocarbon mixtures is identical.

5 Scientific co-worker F. Ya. Molodenko, of the Leningrad State University (LGU) /Leningradskiy Gosudarstvennyy Universitet/, read a paper of I. N. Bushmakin, "Selection of Separation Component (Drainer /uvoditel'/) for the Rectification of Binary Azeotropic Mixtures", and reported on the rectification of binary azeotropic systems with minimum boiling temperature, using as an example the separation of the system formic acid-water. Scientific co-worker N. V. Latugin, of the LGU, presented results of theoretical and experimental studies confirming that the behavior of ternary stratified and unstratified systems when openly vaporized are identical. She indicated that the principal opportunity of separating three-component azeotropes is by means of azeotropic rectification with the addition of a fourth component. Scientific co-worker G. V. Eurova (V. B. Kogan and M. S. Nemtsev), of the VNIISK, made a report on a study of the conditions for the use of separatory agents in the processes of extractive rectification, using as an example a mixture of C<sub>5</sub> hydrocarbons.

Professor L. L. Dobroserdov, of the Voronezh Chemico-Technological Institute, devoted his report to salt rectification, which significantly diminishes the expenditure of heat, the necessary number of plates, and affords a possibility of obtaining final products at a high degree of purity. A paper by Professor E. K. Siyrde, of the Tallinskiy Polytechnic Institute, was concerned with the theory and practice of distillation using water vapor. On the basis of the theoretical analysis of the process and experimental study conducted by him the mechanism of the distillation process was clarified, the process employing water vapor, and optimum conditions for carrying it out were determined. Scientific co-worker V. I. Malyusov, of the Scientific Research Physico-Chemical Institute imeni Kar-pov, reported on the coefficient of separation in the molecular distillation process and on methods of calculating theoretical molecular plates in the process of multi-stage molecular distillation.

Docent Ya. D. Zel'venskiy (V. A. shalygin and Ya. I. Feytik), of the MKhTI, spoke on the advisability of the application of radioactive isotopes for studying the rectification process and for column testing. It is advisable also to standardize experiment and method in evaluating column efficiency, and in studying the rectification process to use diluted solutions which obey Henry's Law.

A considerable number of papers were devoted to techniques of rectification of individual kinds of products. Thus, co-worker V. M. Olevskiy (R. L. Martynenko, L. V. Muzychenco, L. A. Pichugin, Yu. A. Gromoglasov, V. A. Petrov, V. R. Ruchinskiy, and M. Yu. Pogodina), of the GIAP, made a report on the rectification of dimethylterephthal, byproducts of caprolactam synthesis, synthetic cyclohexane, and products of its oxidation. Co-worker of the same institute L. A. Pichugin (I. Ya. Gorodetskiy, R. L. Martynenko, and M. I. Kazymova), reported on the rectificatory purification of waste waters from the production of caprolactam. Scientific co-worker S. M. Danov (G. G. Devyatlykh), of the Scientific Research Institute in the Gor'kiy State University imeni A. A. Zhdanov, reported on the separation of iron chloride and aluminum chloride by rectification. Rectificatory purification of synthetic alcohol up to the condition of food alcohol and the technical certificate was the subject of a paper by scientific co-worker V. P. Gryaznov, of the TsNIISP. Co-worker G. L. Visnevskaya, of the Ukrainian Scientific Research Institute of the Alcohol Industry, made a report on the fractional distillation of molasses alcohol. Docent G. I. Fertman (K. A. Kalunyants), of the All-Union Correspondence Institute of the Food Industry, on the basis of a completed survey and evaluation of existing methods of the operation of beer-rectification apparatus in the food industry, proposed an optimal scheme for the operation of rectification and beer-rectification apparatus. Chief technologist D. A. Kovalenko, of the Andrushevskiy Alcohol Plant, reported on experience in the operation of beer-rectification apparatus.

A number of studies were devoted to problems of phase equilibrium. Scientific co-worker T. A. Pak (V. B. Kogan), of the LIPKh, spoke on methods for the calculations of equilibrium between liquid and vapor in tri-component real systems, which were regarded as quasi-binary. Co-worker I. Ya. Gorodetskiy (V. M. Olevskiy), of the GIAP, presented a paper, "Investigation of the Compressibility of Gases and the Equilibrium of Vapor-Liquid for Thermally Unstable Substances by a Condensation Method." For this study methods and an instrument were developed which made pos-

sible the determination of the temperature of vapor condensation for a known composition from the stream of ionized inert gas.

Docent B. D. Metyushev, of the KTIIPP, presented within broad limits data on phase equilibria between liquid and vapor in the ternary system ethanol-water-higher alcohol. He made a recommendation on the most advisable sites for the separation of higher alcohols (isoamyl, propyl, and isobutyl) from columns.

A paper by co-worker by A. G. Morachevskiy (A. V. Storonkin), of the LGU, was concerned with the question of the composition of binary and ternary azeotropes with changes in temperature and pressure. Thermodynamic equations were derived, which determined the displacement of the composition with changes in temperature for azeotropes of all kinds, which equations were confirmed by experimental data. Co-worker of the same university M. P. Sussarev (A. V. Storonkin) made a report on the differential equations of a family of isocurves /izokribbye/ of relative volatility of tri-component systems, and also indicated the effect of a third component and of the temperature on the composition of a binary azeotrope. Scientific co-worker N. A. Smirnova, of the LGU, presented the results of a thermodynamic investigation of stratified solutions and vapors, on the basis of which she drew the conclusion that the first rule of M. S. Vrevskiy was applicable to systems with a stratified liquid.

In conclusion the conference listened to papers on the automation of rectification assemblies. Co-worker A. M. Popovskiy, of the Experimental Design Bureau of the Committee on Automation, illuminated the basic principles of automatic control of rectification columns and the status of rectification column automation in the chemical industry. Co-worker L. I. Blyakhman (I. I. Zaslavskiy and L. A. Alatyrtsev), of the NIOPiK, spoke on the equations for a continuous-process rectification of a mixture of ethyl alcohol-water by means of a self-adjusting system of automatic search for the optimum regime. Co-worker Yu. A. Alekseyev (Ye. Ya. Susanov), of the Bashkir Affiliate of the SKB - ANN /?/, devoted his paper to a self-adjusting scheme of automatic control of rectification columns, which is a circuit of coupled regulation of the determining parameters for the process and consists of secondary coupled control junctions, holding constant the temperature for the heated vapor and also the constancy of the reflux number.

In its resolutions the conference outlined specific tasks for the future development of the theory and practice of rectification.

The conference regarded as necessary a uniform development of theoretical and experimental studies of the statics of the rectification process (a study of the equilibrium of binary and especially multi-component systems, azeotropic, and extractive rectification) and in kinetics (investigation of the kinetic principles of heat and mass exchange under various hydrodynamic regimes and a study of apparatus hydrodynamics). In order to obtain comparative results of the study of the rectification process and of the testing of rectification apparatus there must be developed standard methods, standard working mixtures and indices; there must be developed approximation modeling methods for rectification apparatus. The conference directed attention to the need to raise theoretical and experimental studies to the point that they can be employed in engineering calculations.

The conference considered as necessary in the field of the development of techniques of rectification and distillation: to search for new design solutions for rectification apparatus which secure cleanliness of separation, high efficiency, and high productivity per unit apparatus volume under various technico-economic indices; to continue the search for the application of the rectification process in new fields (for the production of especially pure metals, metalloids, and salts, for the separation of isotopes, thermally unstable substances, etc.); and to strengthen the development of rational schemes of automation and their introduction into use.

The conference made concrete suggestions in its resolutions on organizational questions for a coordination of scientific research studies in the field of rectification carried out in vuз [plural], scientific research institutes, design offices, and establishments of the chemical, petroleum, and food industries. The conference regarded as essential the organization, in sections on chemical technology and chemical machine construction of the Scientific Technical Council of the Ministry of Higher and Secondary Specialist Education USSR, of a constantly functioning office for rectification, having its duties lying in the responsibility in the coordination of scientific research work, the generalization of results of scientific studies, the development of recommendations for methods of testing rectification columns and for the introduction into industry of results of investigations, and the development of literature searches, the development of a unified terminology, etc.

The conference turned to the Ministry of Higher and Secondary Specialist Education USSR, the Gosplan USSR, and to the Gosplans and Sovnarkhozes of the Union Republics

with a suggestion for the organization in a number of higher educational establishments, field scientific research institutes, and also in separate plants of the chemical and food industries of strengthened experimental equipment for rectification in order to complete laboratory studies on problems of the theory and practice of rectification for purposes of the more rapid introduction of scientific achievements into the public economy.

Secretary of the Organization Committee of the Conference -- P. Tsygankov

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